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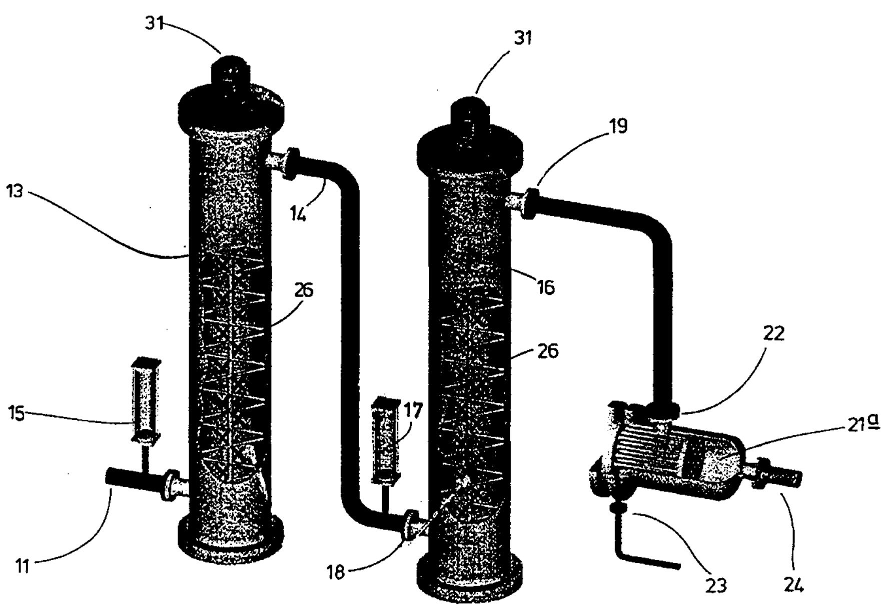
(54) Title: LIQUID–LIQUID SEPARATION



(57) Abstract

A separation process comprising, treating the liquid mixture with one or more additives which promote agglomeration of the droplets of the less dense phase, and then subjecting the treated liquid mixture to separation in a hydrocyclone, the process being characterised in that said treatment includes mechanical agitation of the liquid/additive mixture during flow of the mixture through at least one agglomeration column, the or each column exhibiting a length to diameter ratio not greater than 20:1 and not less than 4:1, and the continuous phase of the liquid mixture having a residence time in the or each column of not less than 15 seconds and not greater than 3 minutes.

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LIQUID-LIQUID SEPARATION

The present invention relates to a process and apparatus for separating, from a mixture, two immiscible liquids of different density, primarily, but not exclusively, oil droplets suspended in a continuous water phase.

It is known to separate immiscible liquids from a mixture by allowing the mixture to remain resident for a predetermined length of time in a gravity separator so that the liquids divide under gravity into a layer of the less dense liquid floating on the surface of a layer of the liquid of greater density.

Another known technique is the use of a hydrocyclone in which the liquid mixture is spun in a vortex within the hydrocyclone such that the more dense liquid forms an outer boundary layer which flows through the cyclone to an underflow outlet while the less dense liquid forms a core which flows in the opposite direction through an overflow outlet of the hydrocyclone. In many applications hydrocyclones are preferred to the use of gravity separators by virtue of the much smaller space occupied for a given processing capacity.

It is recognised however that hydrocyclones are most effective where the droplet size of the less dense liquid is in excess of 15 microns. Where the droplet size is lower than 15 microns the separation efficiency of the hydrocyclone is decreased, and decreases further with decrease in droplet size. An area where particular problems arise is in relation to the processing of produced water from an oil well. Bulk separation techniques of known form will have been used to remove the majority of the oil from the production stream of the oil well leaving produced water to be disposed of. The produced water however will contain oil predominantly in the form of very small droplets, and such produced water must be treated further to

reduce its oil load, ideally to the region of 15 ppm or better, before the produced water can be discharged back into the environment (generally the sea).

It is an object of the present invention to provide a separation process and apparatus for the efficient separation of small droplets of a lighter phase liquid dispersed in a continuous phase of a liquid of greater density.

In accordance with a first aspect of the present invention there is provided a separation process comprising, treating the liquid mixture with one or more additives which promote agglomeration of the droplets of the less dense phase, and then subjecting the treated liquid mixture to separation in a hydrocyclone, the process being characterised in that said treatment includes mechanical agitation of the liquid / additive mixture during flow of the mixture through at least one agglomeration column, the or each column exhibiting a length to diameter ratio not greater than 20:1 and not less than 4:1, and the continuous phase of the liquid mixture having a residence time in the or each column of not less than 15 seconds and not greater than 3 minutes.

In accordance with a further aspect of the present invention there is provided a separation apparatus for the separation of small droplets of a lighter phase liquid dispersed in a continuous phase of a liquid of greater density comprising a treatment column, means for promoting thorough mixing in the column of one or more agglomeration additives and the liquid-liquid mixture to be separated, and a hydrocyclone having its inlet connected to an outlet of said column, whereby liquid mixture flows through said column and into the hydrocyclone wherein the agglomerated droplets of the less dense liquid are separated from the liquid of greater density.

The term “agglomeration” is used herein to denote any process by which the small droplets are brought together in effect to form larger droplets. Thus the droplets may be caused to coalesce so that they genuinely merge to form larger droplets, or the droplets may group together to form larger droplets without actually merging into one another. As a further alternative the droplets may be caused to flocculate by agglomerating onto one another, or onto molecules of a flocculant additive.

In the accompanying drawings:-

Figure 1 is a diagrammatic representation of an apparatus and process in accordance with one example of the present invention,

Figure 2 is a view similar to Figure 1 showing more detail and a minor modification,

Figure 3 is a cross-sectional view of a column of Figure 2,

Figure 4 is a longitudinal sectional view of a fixed part of the column of Figure 3,

Figure 5 is a view on the line 5-5 of Figure 4,

Figure 6 is a longitudinal sectional view of a moving part of the column of Figure 3,

Figure 7 is a cross-sectional view on the line 7-7 of Figure 6, and,

Figures 8 and 9 are longitudinal diagrammatic sectional views of two further column constructions.

Although the example of the invention will be described in relation to the removal of oil droplets from produced water in the treatment of the production flow of an oil well, it is to be recognised that the apparatus and process can be used with other oil / water mixtures, for example bilge water and other mixtures of immiscible liquids of different density.

In Figure 1 of the drawings reference numeral 10 indicates a line carrying produced water from a previous bulk separation process in which the majority of the oil and gas has been separated from the water phase of the well stream. It is desired, particularly in offshore treatment systems, to discharge the produced water back into the ocean, but generally the bulk separation processes leave of the order of 1,000 ppm of oil in the produced water in the form of small oil droplets, many of which may have a diameter below 15 microns. International Standards require a much lower concentration of oil in water before it is permissible to discharge the water back into the environment.

A line 11 takes produced water from the line 10 and feeds it into an agglomeration unit 12 which may take a number of different forms. As drawn the unit 12 includes first and second treatment columns 13, 16 connected in series. The line 11 feeds water into the lower end of the first treatment column 13, the column 13 having an outlet 14 at its upper end. At some point along the length of the column 13, preferably adjacent the inlet from the line 12, is an inlet 15 whereby the oil contaminated water in the column 13 can be dosed with a predetermined chemical additive. The additive is selected in relation to the composition of the mixture in the line

11. Thus, the chemical may be a flocculant additive such as one of a range of known polyelectrolites, which cause flocculation of the oil droplets in the contaminated water. As an alternative, or in addition, the additive could be a coagulant such as ferric chloride and/or a reverse demulsifier polymer, which causes the droplets to coalesce into larger droplets. The objective of the chemical treatment is to agglomerate the small oil droplets suspended in the water phase. Thus the droplets may actually coalesce to form larger droplets, or may group together either binding to one another, or binding to a "skeleton" in the form of molecules of the additives to form flocs. Moreover it will be recognised that additive injection could take place upstream of the column 13 if desired, as shown in Figure 2.

Whatever the agglomeration mechanism, whether it be coalescence, flocculation, or binding together by some other means, the oil droplets which individually are small, group together to form what are in effect larger droplets or particles.

The treated oil and water mixture flowing from the outlet 14 is lead to the inlet 18 at one end of the second, similar treatment column 16 which has an inlet 17 whereby further chemical additives can be introduced into the treated mixture. The additives introduced in the column 16 may be further doses of the same additive introduced in the column 13, or different additives which enhance the agglomerative effect on the oil droplets, and if desired dosing can take place upstream of the inlet 18 as shown in Figure 2.

The outlet 19 of the column 16 is connected through a line to the inlet 22 of a hydrocyclone 21. The hydrocyclone 21 operates in known manner to separate the less dense liquid from the liquid of greater density, the separation efficiency of the hydrocyclone 21 being greatly increased by the

agglomeration of the small oil droplets in the liquid flowing from the column 16. The separated oil leaves the hydrocyclone 21 in known manner through an overflow outlet 23 while the clean water leaves the hydrocyclone through the underflow outlet 24.

In one series of tests, produced water containing oil droplets in the range of 4 to 6 microns was treated by hydrocyclone separation alone which reduced the oil content in the produced water from approximately 1,000 ppm down to between 40 and 80 ppm. Tests using the same produced liquid composition, being treated as described above in a unit 12 followed by a hydrocyclone 21 resulted in the water issuing from the underflow 24 of the hydrocyclone 21 having less than 15 ppm of retained oil.

It will be recognised that in some circumstances the unit may comprise only a single treatment column 13, whereas in other instances it may be beneficial to have two or more treatment columns utilising one, two or more different chemical additives. The term "column" is used herein to refer to a containment vessel of circular cross-section within which chemical/physical treatment of the liquid mixture can occur. The columns 13, 16 are constructed to contain the pressure of the liquid mixture and may be defined by hollow, cylindrical or tapering members of circular cross section, or by pipe loops or by vessels of other shapes. Moreover the columns may be orientated vertically, horizontally, or at some intermediate angle. In each case the or each column will have a length to diameter ratio in the range 4:1 to 20:1, and preferably of the order of 10:1 (the average length to diameter ratio in the case of a tapering column) and the flow through the unit 12 will be such that the continuous phase of the liquid mixture (water in the above example) will have a residence time in the or each column in the range 15 seconds to 3 minutes and preferably 45 seconds to 1 minute.

The point in the or each column at which the dosing additive may be introduced will desirably be at or adjacent the inlet end of the column in relation to the mixture flow in the column, but, as in the Figure 2 construction, the additive inlets 15, 17 could be positioned to inject their additives into the mixture carrying lines upstream of the respective columns 13, 16.

The mixing which takes place within the column or columns is assisted by mechanical agitators 26 in one or more of the columns, which promote a gentle mixing regime, the objective being to increase the contact between the mixture and the additive chemical. The preferred form of agitator is shown in Figures 3 to 7 and has a shaft 27 rotatably driven by an electric motor 31 and extending along the majority of the length of the column 13, 16, the shaft carrying a plurality of outwardly extending vanes 28 which are spaced both axially and circumferentially on the shaft. Desirably the vanes are arranged in axially extending circumferentially spaced rows. The inner wall of the column carries a plurality of stationary vanes 29 or baffles with which the vanes 28 on the shaft 27 cooperate, the stationary vanes 29 being shaped and positioned such that the shaft vanes can pass therebetween. The stationary vanes prevent the mixture circulating in the column with the rotation of the shaft, and thus ensure that the movement of the shaft vanes stirs the mixture. Conveniently the stationary vanes are arranged in axial rows.

As is apparent from Figures 5 and 7 the vanes 29 are equiangularly spaced around the wall of the column but the circumferential arrangement of vanes 28 is such that the vanes 28 are not equiangularly spaced, thereby ensuring that only one axial line of vanes 28 passes through an axial line of vanes 29 at any time.

Desirably the columns 13, 16 and the agitators 26 are formed from a plurality of short standard units a predetermined number of which are assembled end to end to form a column of a desired length.

The rotation of the shaft is controlled to provide tip speed for the shaft vanes in the range 0.3 to 5.0 metres/second, and to minimise the risk of shearing of the agglomerate, but at the same time ensure that efficient mixing of the liquids takes place, the spacing between fixed and moving vanes will not be less than 1.5 mm and will not be greater than 40 mm, preferably in the range 2 to 5 mm. The internal surfaces of the unit 12 can be coated with an oleophilic material to promote agglomeration of a dispersed oil phase on the column wall.

In a modification shown in Figure 8 the column 16 is of smaller diameter than the column 13 and is positioned concentrically within the column 13. One end 14 of the column 13 communicates with the adjacent end 18 of the column 16 and thus the flow through the unit 12 is in one direction along the column 13 and then in the opposite direction along the column 16. The unit inlet is at the end of the column 13 remote from the intercommunicating ends of the columns and the unit outlet 19 is at the adjacent end of the column 16. The dimensions of both columns are within the length to diameter ratio range specified above as are the operating parameters. Only the column 16 is provided with mechanical agitation by means of rotating and fixed vanes 28, 29 as specified above, but additive dosing can be provided for both columns if desired as shown at 15 and 17.

In a further modification as shown in Figure 9 there are two or more columns which are defined by consecutive zones 13, 16 of a single longer vessel. Such an arrangement allows all of the columns to have a vane mixing

mechanism carried by a common motor driven shaft 27 extending longitudinally of the vessel through the consecutive zones thereof.

It will be recognised that irrespective of the exact nature of the unit 12, one or more further units can be connected in parallel with the unit 12 between the line 10 and the hydrocyclone 21 to process greater flow rates. Moreover one or more additional hydrocyclones 21a (Figure 2) can, if needed, be connected in parallel to process the output from the unit 12, or a parallel array of units 12.

Control systems can be utilised to determine and control the dosing of the additive chemicals in accordance with the composition and flow rate of the produced water in the line 12, and if necessary a pump arrangement can be provided to achieve the necessary pressure in the system to achieve the desired liquid flow.

The system can be pressurised from the line 10 provided of course that sufficient pressure is available in the dosing injection system at the inlets 15 and 17 to permit dosing chemicals to be introduced. The unit 12 can be arranged to produce a pressure drop, in relation to the pressure in the line 10, such that treated liquid enters the hydrocyclone inlet 22 at optimum pressure and flow rates in relation to the operating conditions of the hydrocyclone 21. Moreover the connection between the outlet of the unit or units 12 and the or each hydrocyclone 21 is arranged to cause minimal pressure drop, desirably not exceeding 1 bar, so the there is minimal shearing of the agglomerate in its passage from the unit(s) 12 to the hydrocyclone(s) 21.

The method and apparatus described above is particularly suited for use in the oil industry on a floating production vessel since it is substantially impervious to motion, the hydrocyclone being insensitive to its orientation.

CLAIMS

1. A process for the separation of small droplets of a lighter phase liquid dispersed in a continuous phase of a liquid of greater density, the process comprising, treating the liquid mixture with one or more additives which promote agglomeration of the droplets of the less dense phase, and then subjecting the treated liquid mixture to separation in a hydrocyclone, the process being characterised in that said treatment includes mechanical agitation of the liquid / additive mixture during flow of the mixture through at least one agglomeration column, the or each column exhibiting a length to diameter ratio not greater than 20:1 and not less than 4:1, and the continuous phase of the liquid mixture having a residence time in the or each column of not less than 15 seconds and not greater than 3 minutes.

2. A process as claimed in Claim 1, characterised in that the length to diameter ratio of the or each column is of the order of 10:1.

3. A process as claimed in Claim 1 or Claim 2, characterised in that said residence time in the or each column is not less than 45 seconds and not greater than 1 minute.

4. A process as claimed in any one of the preceding claims, characterised in that one of said one or more additives effects agglomeration of said droplets by coalescence.

5. A process as claimed in Claim 4, characterised in that said additive includes Ferric Chloride.

6. A process as claimed in Claim 4 or Claim 5, characterised by a further additive treatment effecting flocculation.

7. A process as claimed in Claim 6, characterised in that said additive includes a polymer.

8. A process as claimed in any one of the preceding claims, characterised by subjecting said mixture to mechanical agitation throughout the majority of the length of the or each column.

9. A process as claimed in any one of the preceding claims, characterised in that said mechanical agitation is effected by a shaft carrying outwardly extending vanes, the assembly of shaft and vanes being rotated about the shaft axis in said column.

10. A process as claimed in Claim 9, characterised by the provision of fixed vanes in said column with which said rotating vanes coact.

11. A process as claimed in Claim 10, characterised by a rotational speed, measured at the outer tips of the rotating vanes in the range 0.3 to 5.0 m/s.

12. A process as claimed in Claim 10 or Claim 11, characterised in that the gap between fixed and moving parts of the mechanical agitation mechanism is not less than 2 mm and not greater than 5 mm.

13. A process as claimed in any one of the preceding claims, characterised by a pressure drop not exceeding 1.0 bar in between the treatment unit outlet and the hydrocyclone inlet.

14. A separation apparatus for the separation of small droplets of a lighter phase liquid dispersed in a continuous phase of a liquid of greater density comprising a treatment column, means for promoting thorough mixing in the column of one or more agglomeration additives and the liquid-liquid mixture to be separated, and a hydrocyclone having its inlet connected to an outlet of said column, whereby liquid mixture flows through said column and into the hydrocyclone wherein the agglomerated droplets of the less dense liquid are separated from the liquid of greater density.

15. Apparatus as claimed in Claim 12 or Claim 13, characterised in that additive treatment takes place in two or more columns in series with one another.

16. Apparatus as claimed in Claim 14 or Claim 15, characterised by the or each column exhibiting a length to diameter ratio not greater than 20:1 and not less than 4:1.

17. Apparatus as claimed in Claim 16, characterised in that a second column is positioned within a first column.

18. Apparatus as claimed in Claim 17, characterised in that said columns are of circular cross-section and said second column is arranged concentrically within said first column with its inlet end adjacent the outlet end of the first column such that the flow through the second column is in a direction opposite that in the first column.

19. Apparatus as claimed in any one of Claims 14 to 18, characterised in that internal surface of the or each column is oleophilic.

20. Apparatus as claimed in Claim 15, characterised in that said columns are defined by consecutive zones of a single longer column.

21. Apparatus as claimed in any one of Claims 14 to 20, characterised in that said means for promoting thorough mixing comprises a shaft carrying outwardly extending vanes, the assembly of shaft and vanes being rotated about the shaft axis in said column to effect mechanical agitation of the liquid.

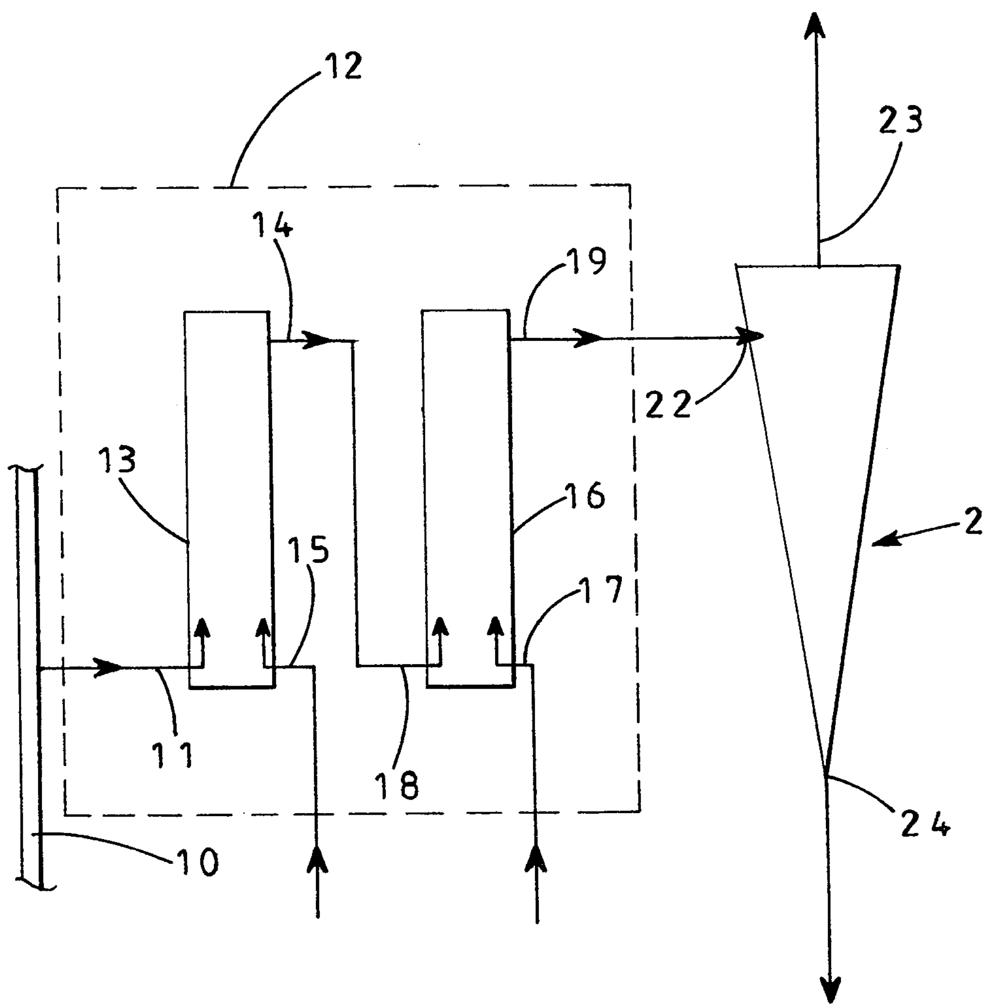

FIG 1

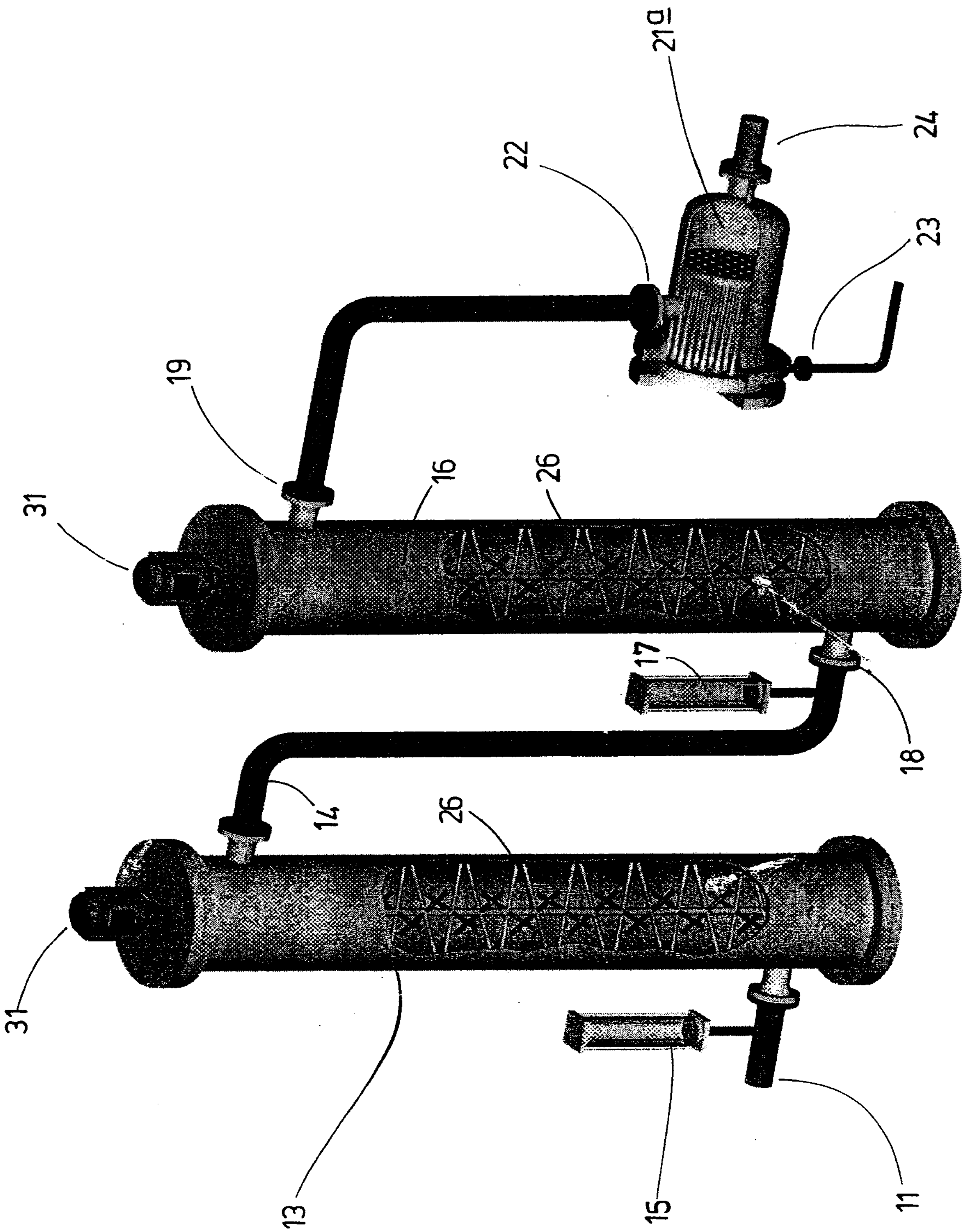

FIG 2

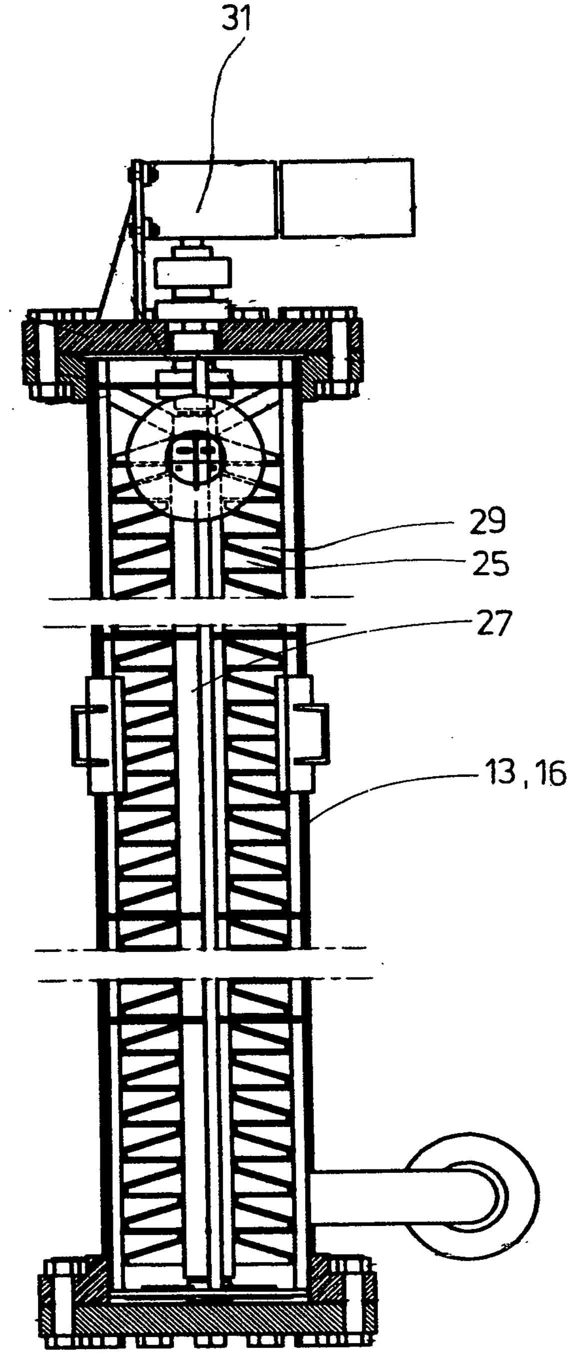

FIG 3

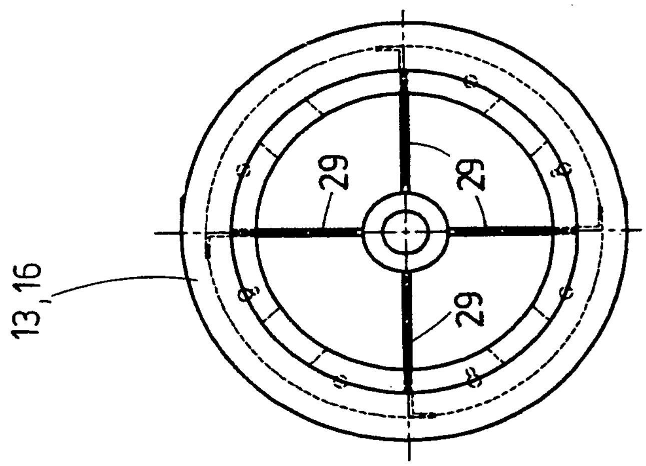

FIG 5

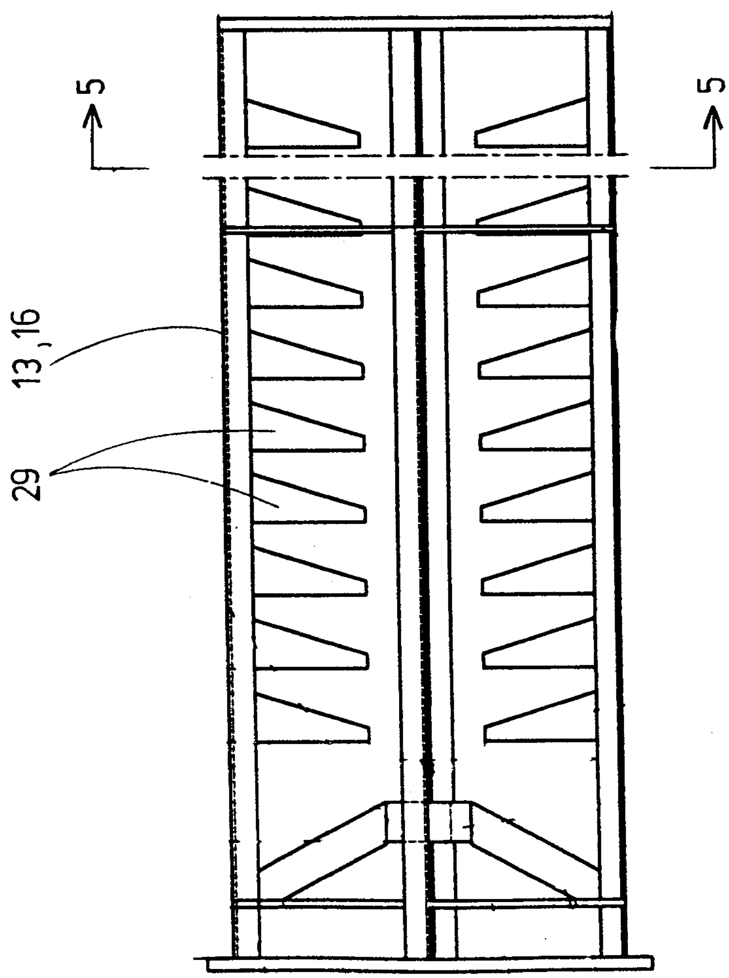

FIG 4

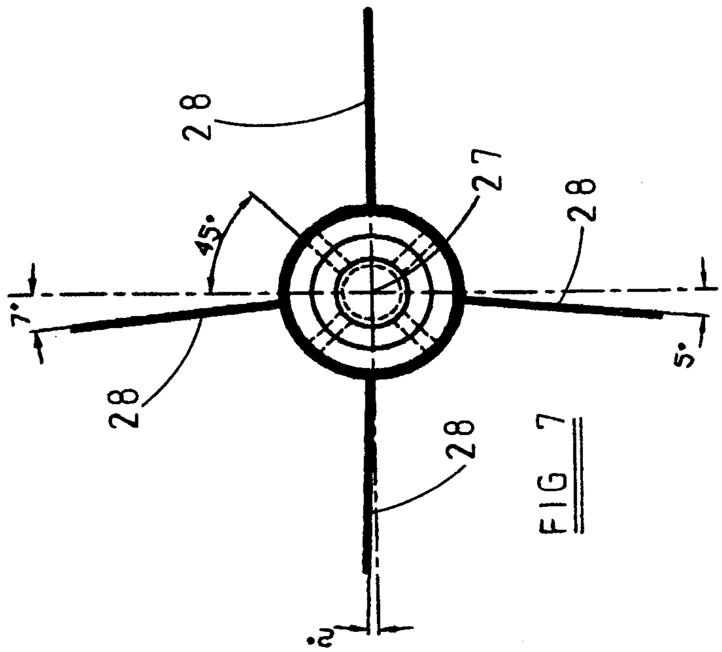

FIG 7

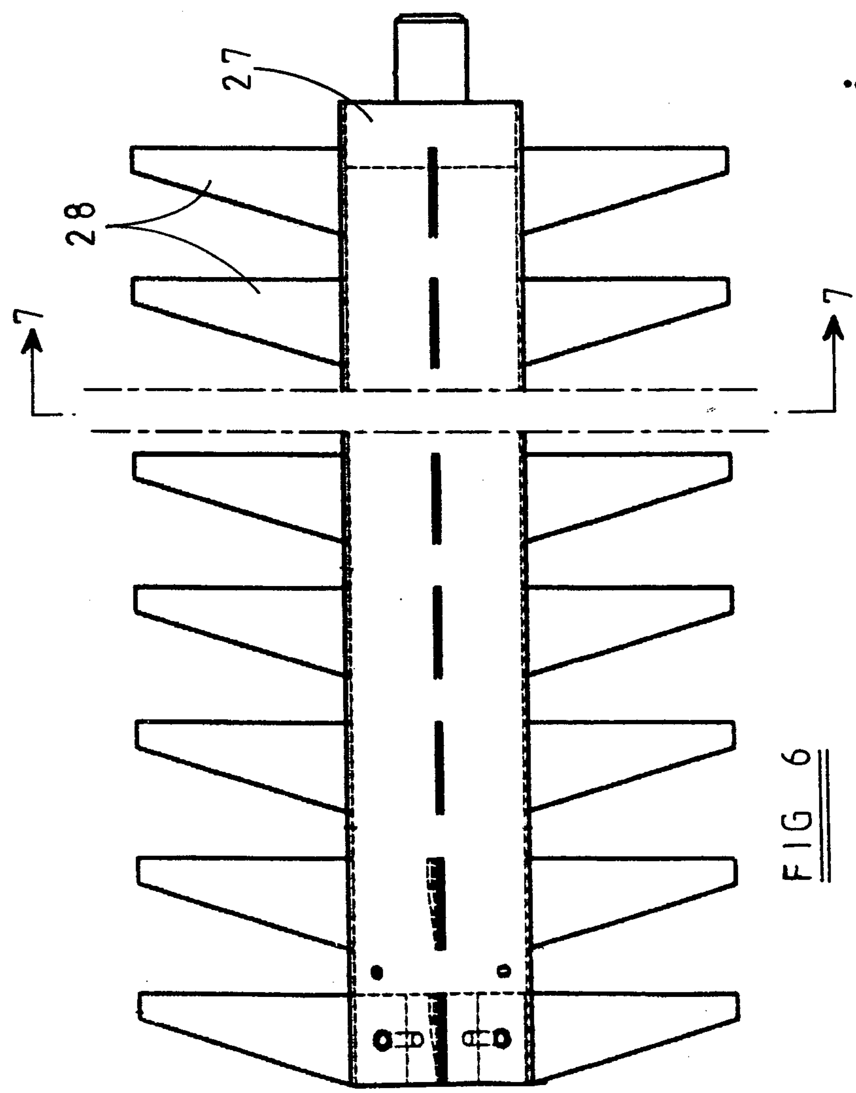

FIG 6

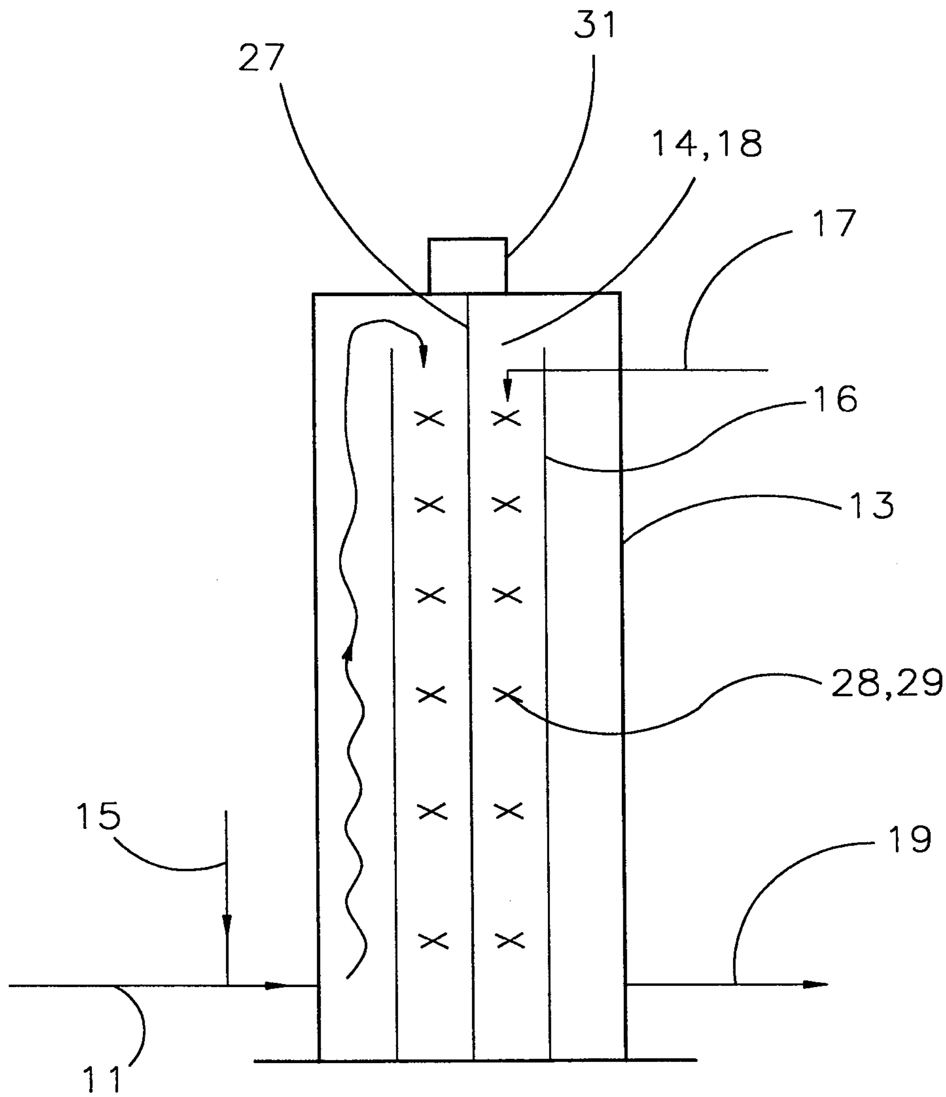

FIG 8

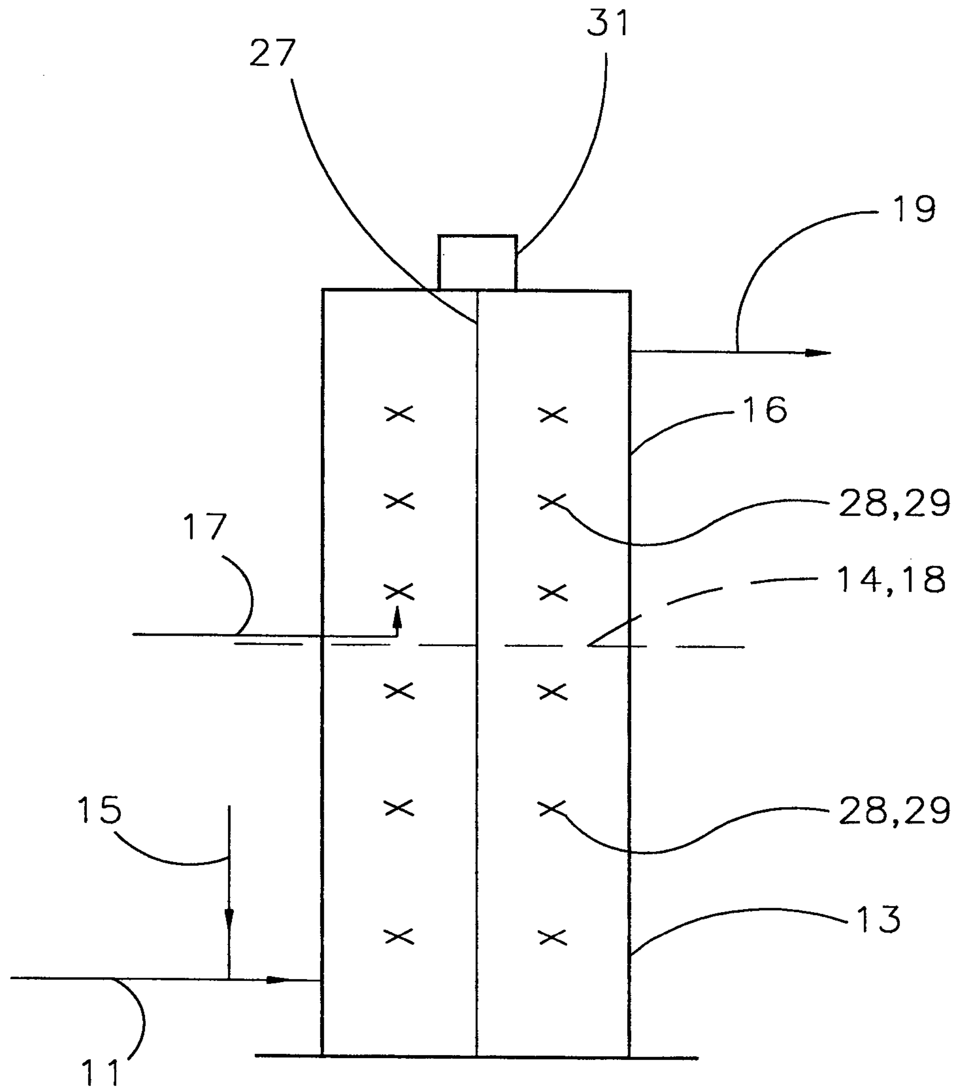

FIG 9

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/02564

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B01D17/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 37 12 106 A (RWO MASCHINENFABRIK) 20 October 1988 (1988-10-20) claim 1; figure 1 ---	1,14
A	FR 1 345 193 A (SHELL INTERNATIONAL RESEARCH MAATSCHAPPIJ) 28 October 1963 (1963-10-28) claim 1; figures 1,2 ---	1,14
A	US 2 881 920 A (D.J.SIMKIN) 14 April 1959 (1959-04-14) the whole document ---	1,14
A	US 2 698 303 A (C.M.BLAIR, JR. ET AL.) 28 December 1954 (1954-12-28) claim 1; figures 3,8 ---	14,17,18
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
13 December 1999	20/12/1999

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/02564

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
A	DD 108 657 A (R.ELSPASS ET AL.) 5 October 1974 (1974-10-05) the whole document -----	1

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INTERNATIONAL SEARCH REPORT

Information on patent family members

Interr   ıal Application No

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